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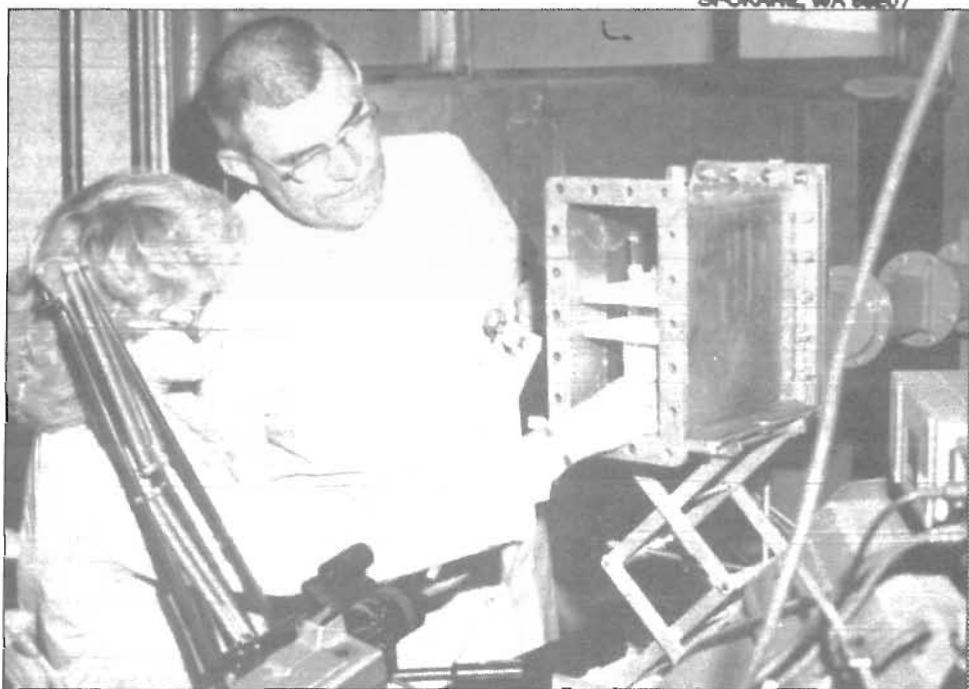
## Microwave Heating of Chemicals and Minerals

By S. L. McGill, J. W. Walkiewicz, and A. E. Clark

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*Cover: Scientists at the USBM Reno Research Center determining the microwave heating characteristics of minerals.*

**Report of Investigations 9518**

# **Microwave Heating of Chemicals and Minerals**

**By S. L. McGill, J. W. Walkiewicz, and A. E. Clark**

**UNITED STATES DEPARTMENT OF THE INTERIOR  
Bruce Babbitt, Secretary**

**BUREAU OF MINES**

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## CONTENTS

	<i>Page</i>
Abstract .....	1
Introduction .....	2
Experimental techniques .....	2
Equipment .....	2
Materials .....	3
Procedure .....	3
Results and discussion .....	4
Fixed power study .....	4
Variable power study .....	8
Summary .....	10
References .....	11
Appendix A.—Descriptive list of the microwaved samples .....	12
Appendix B.—Tyler sieve screen analyses of chemicals and minerals microwaved in the varied power level study .....	15
Appendix C.—Heating-rate curves of samples microwaved in the varied power level study .....	16

## ILLUSTRATIONS

1. Fixed power 1,000-W microwave oven with thermocouple inserted into the sample test cell .....	3
2. Applicator used for variable power studies with thermocouple inserted into sample test cell .....	3
3. Microwave-induced stress cracking in a taconite (iron ore) sample .....	9
4. Microwave heating-rate curves .....	10

## TABLES

1. Effect of fixed power microwave heating on the temperature of reagent-grade elements and compounds ..	5
2. Effect of fixed power microwave heating on the temperature of natural minerals .....	7
3. Effect of incident microwave power on the heating rates of selected chemicals and minerals .....	7

### UNIT OF MEASURE ABBREVIATIONS USED IN THIS REPORT

cm      centimeter

g        gram

MHz    megahertz

min     minute

mL      milliliter

pct     percent

s        second

W       watt

°C      degree Celsius

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# MICROWAVE HEATING OF CHEMICALS AND MINERALS

By S. L. McGill,<sup>1</sup> J. W. Walkiewicz,<sup>2</sup> and A. E. Clark<sup>1</sup>

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## ABSTRACT

The U.S. Bureau of Mines has determined the microwave heating rates for a suite of minerals and reagent-grade compounds. Maximum temperature achieved for the samples and the time required to reach temperature are reported. Heating rates of powdered samples are presented as a function of incident powers up to 3,000 W at 2,450 MHz. Descriptions of the microwave cavities, test cells, and a thermocouple assembly used to measure temperature in the microwave field are given. Many minerals of value heated in the microwave field, whereas host rock or gangue materials did not. In general, heating rates increased as incident power was increased. Exceptions to this were some very good (CuO, Fe<sub>3</sub>O<sub>4</sub>, and PbS) and very poor (CaCO<sub>3</sub> and SiO<sub>2</sub>) microwave-absorbing materials that showed negligible changes with increased power. Applications demonstrating the use of microwave energy in mineral processing that are described herewith include oxidation-reduction reactions, mineral liberation, and mercury retorting-vaporization. Microwave data on mineral heating rates will provide insight to possible chemical and mineral processing applications as well as assist in predictions of processing parameters.

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## INTRODUCTION

Microwave technology has been used extensively and economically in many commercial applications such as cooking, thawing, drying, and vulcanizing (1-2).<sup>3</sup> Microwave heating has distinct advantages when compared with conventional heating. Microwaves can penetrate a material and heat the interior directly. Selective heating is possible since the ability to absorb microwaves differs from one material to another. In highly absorbing materials, microwaves can produce very rapid heating rates. The unique properties of microwaves offer the potential for applications in extractive processing not possible with conventional heating. Possible mineral processing applications include expanding, sintering, melting, and vaporizing. Microwave-induced chemical reactions such as oxidation, reduction, exchange, and polymerization may also be possible. Metaxas and Meredith (3) have reported the use of microwaves in promoting chemical reactions such as ferromagnetization of pyrite, devulcanization of scrap rubber, and cracking of waste plastics.

Since microwaves are not used widely in the minerals industry, little data have been published on the microwave-heating characteristics of minerals and compounds. Chen and others (4) described the general behavior of a number of minerals that were exposed to microwave radiation in air at 2,450 MHz; their results were qualitative and descriptive in nature. Standish and others (5) investigated mathematical modeling for predicting temperature

distribution in a dry dielectric material subjected to microwave heating (2,450 MHz) using iron ore and carbon compounds. Dielectric properties of minerals at elevated temperatures were reported by Holderfield and Salsman (6). Microwave assisted drilling in hard rock has been investigated by Lindroth and others (7). Many exploratory studies and experiments have been performed on the microwave heating and processing of ceramics and composite materials as reported by Sutton (8).

This paper describes U.S. Bureau of Mines (USBM) research to identify potential applications using microwaves for mineral processing based on the heating responses of chemicals and minerals. This research is part of the USBM's mission to enhance efficiency in the production of domestic minerals.

Microwave heating rates measured with a sheathed thermocouple are reported for selected chemicals and minerals at varied incident power levels. Heating rates were determined at fixed power (275 W) in a preliminary study and at varied power levels (500 to 3,000 W) in a detailed investigation. Our research has shown that many minerals of value, such as galena, ilmenite, magnetite, and chalcopyrite, absorbed microwaves and were heated, whereas most common host rock minerals, such as quartz, calcite, and feldspar, were not heated. This selective heating by microwaves has potential extractive metallurgy applications that are illustrated in this report.

## EXPERIMENTAL TECHNIQUES

### EQUIPMENT

Preliminary microwave heating studies were conducted in a fixed power 1,000-W, 2,450-MHz Sharp model R22AC commercial oven (figure 1). A vented 1,000-mL water load was placed in the oven chamber to prevent excess reflection to the power tube. For the varied power level study, the microwave source was a variable power (up to 3,000 W), 2,450-MHz Cober microwave unit, model 53. A modified WR 975 rectangular waveguide (29.85 by 12.38 by 24.77 cm) served as an applicator (oven or chamber) that was coupled to the power source using WR 284 waveguides (figure 2). Included in the waveguide sections were a protective circulator that directed reflected power into a dummy water load, and a triple-stub tuner used to match impedance between the source and sample. The waveguide was coupled to the applicator such that the sample was along the geometric center line of incident microwave

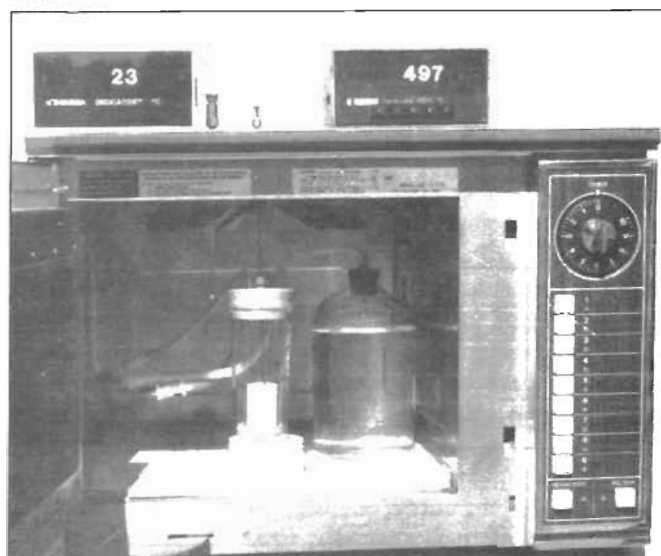
irradiation. The chamber was equipped with perforated screen windows for video viewing during testing. All applicators used for the preliminary fixed power and the varied power study were equipped with ports for inlet and outlet gas lines.

A method for continuously monitoring sample temperatures was designed (9). A sheathed thermocouple was inserted through the roof of the chamber directly into the sample, as shown in figures 1 and 2. The thermocouple was type K with an ungrounded tip sheathed in Inconel 702 alloy (for sulfide samples the sheath was 440 stainless steel). The accuracy of the thermocouple data was within  $\pm 2$  pct, and was determined from measurements made on boiling water and applied to samples that absorb microwaves. For samples considered to be microwave transparent (heat to less than 100 °C), an error greater than  $\pm 2$  pct could be expected because microwaves could penetrate the sample and heat the oxidized surface of the thermocouple sheath. Sample temperature was recorded continuously with a strip-chart recorder during irradiation.

<sup>3</sup>Italic numbers in parentheses refer to items in the list of references preceding the appendix.



Figure 1



*Fixed power 1,000-W microwave oven with thermocouple inserted into the sample test cell.*

The sample test cell that was used in the preliminary fixed power experiments consisted of a nominal 20-mL alumina crucible placed on a ceramic insulating brick inside a 250-mL Pyrex glassware beaker. The beaker was sealed with a microwave-transparent gum rubber stopper fitted with ports for gas flow and the thermocouple. Although this test cell was adequate for the fixed power study, the Pyrex glassware and gum rubber materials were not suitable at the higher power levels. Therefore, a controlled-atmosphere, microwave-transparent test cell compatible with power levels encountered using the 3,000-W microwave power source was fabricated from Teflon nonstick coating and fused quartz. The cell consisted of a 10.16-cm-OD, 15.24-cm-high fused quartz tube fitted between two (square) 11.75-cm grooved Teflon nonstick coated plates and secured using threaded Teflon nonstick coated rods. Thermocouple and inlet and outlet gas flow ports were installed in the top plate. A 9.21-cm-diam, 6.05-cm-high cylinder of insulation brick centered inside the quartz tube protected the Teflon nonstick coated base from the heat generated in the sample. Each sample was placed in an alumina crucible that was centered in a 2.54-cm-deep, 2.86-cm-diam well in the microwave-transparent insulation brick.

## MATERIALS

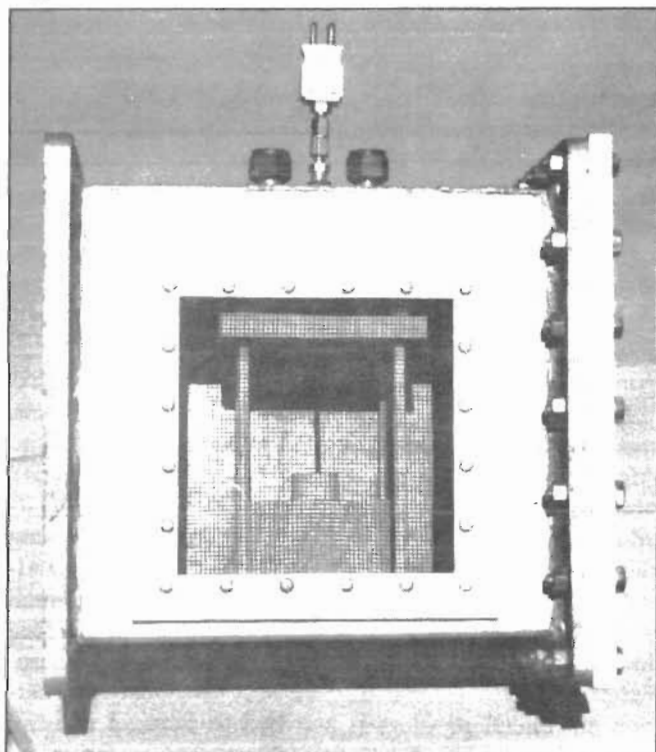
Reagent-grade chemicals and high-purity minerals were selected for the investigation. In the fixed power study, all samples were powders except for the three liquids, Hg,  $\text{SnCl}_4$ , and  $\text{TiCl}_4$ . A description of the elements and compounds is given in appendix A. The commercial source, chemical grade, and purity are reported according to the data available on the container label. Grade and purity were not indicated by all manufacturers. The minerals tested were obtained from various sources and were identified by X-ray diffraction analysis. These samples were high-grade specimens with a minimum purity of 90 pct. Particle size distribution was not determined for samples tested at the fixed power level, but no particle size exceeded 32 mesh.

In the varied power study, minerals and chemicals commonly found in ores or used in mineral processing were selected. The compounds tested were generally from the same source identified in appendix A. If the material was no longer available, a sample of equivalent grade or purity was tested. For this study, particle size distributions were determined by dry screening tests using Tyler sieves and wet screening for clayey materials. The data are given in appendix B.

## PROCEDURE

For the fixed power study, samples of 25 g or a constant volume of 18 mL for low bulk density materials were

Figure 2



*Applicator used for variable power studies with thermocouple inserted into sample test cell.*

placed in the 20-mL alumina crucible. The sample was handtapped until no further settling was observed. The crucible was placed inside a Pyrex glassware beaker that was capped with a rubber stopper and was precisely positioned in the microwave oven. The thermocouple was inserted into the center of the sample and argon gas was introduced to the cell. The samples were generally irradiated for an arbitrarily chosen 7 min during which time temperature was continuously monitored and recorded.

For the varied power study, sample sizes were 25 to 50 g for high-bulk density materials or volumes of 12 to 14 mL for low-density materials. No effect of sample mass or volume on the heating rate was observed over the range tested due to the limited sensitivity of the thermocouple. A 15.5-mL alumina crucible containing the sample was placed in the Teflon nonstick coated and quartz cell that was identically positioned in the chamber for each test. All samples were irradiated under an argon atmosphere with a thermocouple in place for continuous monitoring of temperature.

To maximize heating, the impedance was matched between the power source and the sample using a triple stubtuner. Three mineral samples ( $\text{SiO}_2$ ,  $\text{ZnO}$ , and  $\text{Fe}_3\text{O}_4$ ), which represent poor, medium, and good

absorbers, were used for tuning. Matching was accomplished with tuner adjustments using microwave-absorbing neon bulbs as the primary indicator. Neon bulbs (types NE76, NE81, and NE97) were slightly embedded in the samples prior to irradiation. The triple stub tuner was adjusted until the neon bulb was lit at a minimum incident power (approximately 50 W). With the neon bulb removed and the thermocouple in place, heating rates and forward and reflected power measurements (Boonton model 4200 power meters) confirmed the impedance matching. Heating data were collected with no additional tuning between tests. Samples were tested at 500-W increments ranging from 500 to 3,000 W. Whenever possible, tests were run for 7 min in both the fixed power study and the varied power study. However, the tests were terminated when melting, boiling, or arcing was observed or when the temperature stabilized, decreased, or approached the thermocouple limit. A decrease in sample temperature was indicative that some portion of the test cell was beginning to absorb microwaves. This would cause unreliable sample heating-rate data and possibly destroy the cell. For highly absorbing samples, the test was discontinued when the temperature reached 1,100 °C to prevent melting of the thermocouple sheath.

## RESULTS AND DISCUSSION

Microwave heating rates for selected chemicals and minerals are presented for the fixed power study in tables 1 and 2, and for the varied power study in table 3. In tables 1 and 2, the sample temperatures obtained at 1 min are shown. The maximum temperature and the time interval to attain maximum temperature from ambient temperature (20° to 30 °C) are shown in tables 1, 2, and 3. Time values are included so that a comparison of heating-rate data can be made.

A basis for comparison between the microwave heating data obtained with the fixed power Sharp microwave oven containing a 1,000-mL water load and data obtained with the variable power Cober microwave apparatus was established. In the 1,000-W Sharp oven with the water load present, the actual power level incident upon the sample can not be determined precisely. The amount of microwave energy absorbed by the water load was not a constant and varied according to the heating rate of the sample. More energy was absorbed by the water when the sample was a poor absorber. Heating-rate curves were determined for a 15-g deionized water sample at power levels of 200, 300, 400, and 500 W in the Cober microwave applicator. These data were compared to the heating-rate curves for water obtained in the Sharp oven with the 1,000-mL water load in place. Cober microwave heating rates similar to the Sharp oven heating rates were obtained at a power level between 200 and 300 W. From an

extrapolation based on the slopes of the heating-rate curves, it was determined that a Cober microwave power level of  $275 \pm 25$  W is equivalent to the 1,000-W Sharp microwave oven containing a 1,000-mL water load.

### FIXED POWER STUDY

The highest temperatures were obtained with the metal oxides  $\text{NiO}$ ,  $\text{MnO}_2$ ,  $\text{Fe}_3\text{O}_4$ ,  $\text{Co}_2\text{O}_3$ ,  $\text{CuO}$ , and  $\text{WO}_3$  (tables 1 and 2). Most metal sulfides heated well but showed no obvious pattern of microwave heating. Some samples exhibited an extremely rapid rate of heating at the fixed (275-W) power level. The powdered amorphous carbon sample heated to 1,283 °C in 1 min and was typical of the microwaved amorphous carbon. The fastest heating rate obtained with amorphous carbon was 38 °C/s, which heated from an ambient temperature of 29° to 1,170 °C during the first 30 s and to a maximum of 1,234 °C at 1 min. Although powdered carbon absorbs microwaves, solid carbon such as a crucible reflects microwaves. The same is true for metal powders. Although solid metals reflect microwaves, the metal powders heated well. The heating of carbon and metal powders is ascribed to induced resistive heating in conducting materials. The anhydrous chlorides did not heat above 112 °C except for  $\text{SnCl}_2$ ,  $\text{CuCl}$ , and  $\text{ZnCl}_2$ . Common gangue minerals such as quartz and feldspars (albite and orthoclase) did not heat.

**Table 1.—Effect of fixed power microwave heating on the temperature of reagent-grade elements and compounds**

Chemical	Temp, °C at 1 min	Maximum temp, °C	Time, min
AgBr	37	39	1.5
AgCl	39	45	4
AgI	51	64	4
Ag <sub>2</sub> S	422	652	5.25
Al	380	577	6
AlCl <sub>3</sub>	35	41	4
AlCl <sub>3</sub> •6H <sub>2</sub> O	36	46	4
Al(ClO <sub>3</sub> ) <sub>3</sub> •6H <sub>2</sub> O	48	72	5
Al <sub>2</sub> O <sub>3</sub>	55	78	4.5
Al <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> •18H <sub>2</sub> O	116	206	3.75
Al <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> •K <sub>2</sub> SO <sub>4</sub> •24H <sub>2</sub> O	46	56	4.5
BaCl <sub>2</sub>	37	45	4
BaCl <sub>2</sub> •2H <sub>2</sub> O	50	50	0.75
BaS	110	160	5
C (amorphous, <1 μm)	1,283	1,283	1
C (graphite, <1 μm)	598	1,073	1.75
C (graphite, -200 mesh)	409	780	6
CaBr <sub>2</sub> •2H <sub>2</sub> O	37	47	4
CaCl <sub>2</sub>	31	32	1.75
CaCl <sub>2</sub> •2H <sub>2</sub> O	45	45	0.75
CaCO <sub>3</sub>	41	54	7
CaF <sub>2</sub>	66	81	4.25
CaO	73	116	4
CaS	66	102	4.5
CCl <sub>4</sub>	28	34	4
CdCl <sub>2</sub>	31	37	4
CdCl <sub>2</sub> •2.5H <sub>2</sub> O	134	144	2.5
CdS	70	87	5
CeCl <sub>3</sub>	40	48	2.75
CeCl <sub>3</sub> •7H <sub>2</sub> O	30	38	1.5
CeO <sub>2</sub>	74	114	4.5
Co	687	697	3
CoCl <sub>2</sub>	34	36	2
CoCl <sub>2</sub> •6H <sub>2</sub> O	112	153	4
Co <sub>2</sub> O <sub>3</sub>	724	1,290	3
CoS	74	158	7
CrCl <sub>3</sub>	67	82	6
CrCl <sub>3</sub> •6H <sub>2</sub> O	93	181	5
CrO <sub>3</sub>	74	112	9
Cr <sub>2</sub> O <sub>3</sub>	65	130	7
Cu	120	228	7
CuBr	503	722	11
CuBr <sub>2</sub>	140	193	3.25
CuCl	38	619	13
CuCl <sub>2</sub> •2H <sub>2</sub> O	147	171	2.75
CuF <sub>2</sub> •2H <sub>2</sub> O	56	78	6
CuO	344	1,012	6.25
Cu <sub>2</sub> O	50	89	7
CuS	154	440	4.75
Fe	681	768	7
FeBr <sub>2</sub> •6H <sub>2</sub> O	64	180	4
FeCl <sub>2</sub>	32	33	1.5
FeCl <sub>2</sub> •2H <sub>2</sub> O	34	41	4
FeCl <sub>2</sub> •4H <sub>2</sub> O	39	47	4
FeCl <sub>3</sub>	32	41	4
FeCl <sub>3</sub> •6H <sub>2</sub> O	59	220	4.5
Fe <sub>2</sub> O <sub>3</sub>	38	58	7
Fe <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> •9H <sub>2</sub> O	96	154	6
Hg	28	40	6
HgCl <sub>2</sub>	80	112	7
HgS	70	105	7
KBr	47	49	0.25
KCl	31	31	1
LaCl <sub>3</sub>	36	38	1.75

Table 1.—Effect of fixed power microwave heating on the temperature of reagent-grade elements and compounds—Continued

Chemical	Temp, °C at 1 min	Maximum temp, °C	Time, min
LaCl <sub>3</sub> •7H <sub>2</sub> O	32	34	2
La <sub>2</sub> O <sub>3</sub>	86	119	6.5
LiBr•H <sub>2</sub> O	45	45	1
LiCl	35	35	0.5
Mg	85	120	7
MgBr <sub>2</sub> •6H <sub>2</sub> O	90	322	9
MgCl <sub>2</sub> •6H <sub>2</sub> O	149	254	4
MgO	138	203	5.5
MgSO <sub>4</sub> •7H <sub>2</sub> O	57	80	4.5
MnBr <sub>2</sub> •4H <sub>2</sub> O	165	176	2.25
MnCl <sub>2</sub>	52	53	1.75
MnCl <sub>2</sub> •4H <sub>2</sub> O	130	170	4
MnO <sub>2</sub>	794	1,287	6
MnSO <sub>4</sub> •H <sub>2</sub> O	37	47	5
Mo	531	660	4
MoO <sub>3</sub>	48	69	5.5
NaBr	34	40	4
NaCl	57	83	7
Na <sub>2</sub> MoO <sub>4</sub>	41	53	4.25
Na <sub>2</sub> SO <sub>4</sub>	47	67	6
Nb	91	358	6
Nb <sub>2</sub> O <sub>5</sub>	67	114	6
NH <sub>4</sub> Cl	30	31	3.5
5(NH <sub>4</sub> ) <sub>2</sub> •12WO <sub>3</sub>	52	68	4
Ni	384	384	1
NiBr <sub>2</sub> •2H <sub>2</sub> O	110	159	3
NiCl <sub>2</sub>	42	51	2.75
NiCl <sub>2</sub> •6H <sub>2</sub> O	163	168	1.75
NiF <sub>2</sub> •4H <sub>2</sub> O	41	50	5
NiO	336	1,305	6.25
NiS	176	251	7
Pb	186	277	7
PbBr <sub>2</sub>	51	56	2.75
PbCl <sub>2</sub>	48	51	2
PbO	78	91	2.25
PbS	955	1,024	1.25
PrCl <sub>3</sub>	29	30	1.25
S	97	163	6
Sb	390	390	1
SbCl <sub>3</sub>	199	224	1.75
SbCl <sub>5</sub>	31	54	6
Sn	144	297	6
SnBr <sub>2</sub>	378	472	10
SnCl <sub>2</sub>	59	476	2
SnCl <sub>2</sub> •2H <sub>2</sub> O	34	252	5.25
SnCl <sub>4</sub>	30	49	8
SnCl <sub>4</sub> •5H <sub>2</sub> O	55	151	6.75
SrCl <sub>2</sub> •6H <sub>2</sub> O	30	32	3
Ta	89	177	7
TiB <sub>2</sub>	530	843	7
TiCl <sub>4</sub>	26	31	4
TiO <sub>2</sub>	46	79	8.5
V	557	557	1
V <sub>2</sub> O <sub>5</sub>	288	714	11
YCl <sub>3</sub>	37	40	1.75
Y <sub>2</sub> O <sub>3</sub>	76	115	7
W	468	690	6.25
WCl <sub>6</sub>	41	45	3
WO <sub>3</sub>	95	1,270	6
Zn	505	581	3
ZnBr <sub>2</sub>	108	574	7
ZnCl <sub>2</sub>	232	609	7
ZnO	60	326	5.5
ZnS	62	67	3
Zr	260	462	6
ZrO <sub>2</sub>	49	63	4

Table 2.—Effect of fixed power microwave heating on the temperature of natural minerals

Mineral	Chemical composition	Temp, °C at 1 min	Maximum temp, °C	Time, min
Albite	NaAlSi <sub>3</sub> O <sub>8</sub>	53	82	7
Arizonite	Fe <sub>2</sub> O <sub>3</sub> •3TiO <sub>2</sub>	84	290	10
Chalcocite	Cu <sub>2</sub> S	506	746	7
Chalcopyrite	CuFeS <sub>2</sub>	920	920	1
Chromite	FeCr <sub>2</sub> O <sub>4</sub>	89	155	7
Cinnabar	HgS	83	144	8
Galena	PbS	755	956	7
Hematite	Fe <sub>2</sub> O <sub>3</sub>	102	182	7
Magnetite	Fe <sub>3</sub> O <sub>4</sub>	861	1,258	2.75
Marble	CaCO <sub>3</sub>	50	74	4.25
Molybdenite	MoS <sub>2</sub>	108	192	7
Orpiment	As <sub>2</sub> S <sub>3</sub>	71	92	4.5
Orthoclase	KAlSi <sub>3</sub> O <sub>8</sub>	49	67	7
Pyrite	FeS <sub>2</sub>	816	1,019	6.75
Pyrrhotite	Fe <sub>1-x</sub> S	730	886	1.75
Quartz	SiO <sub>2</sub>	52	79	7
Sphalerite	ZnS	65	87	7
Tetrahedrite	Cu <sub>12</sub> Sb <sub>4</sub> S <sub>13</sub>	64	151	7
Zircon	ZrSiO <sub>4</sub>	41	52	7

Table 3.—Effect of incident microwave power on the heating rates of selected chemicals and minerals<sup>1</sup>

Chemical	Wt, g	Vol, mL	500 W		1,000 W		1,500 W		2,000 W		2,500 W		3,000 W	
			Temp, °C	Time, min	Temp, °C	Time, min	Temp, °C	Time, min	Temp, °C	Time, min	Temp, °C	Time, min	Temp, °C	Time, min
Al <sub>2</sub> O <sub>3</sub> (α)	14.0	14	385	7	463	7	498	7	547	7	>1,200	<sup>2</sup> 6.67	>1,200	<sup>2</sup> 3.25
CaCl <sub>2</sub>	17.0	14	35	7	43	7	53	7	58	7	37	<sup>3</sup> 0.75	-	-
CaCO <sub>3</sub>	10.0	14	32	7	42	7	51	7	65	7	-	-	-	-
Cr <sub>2</sub> O <sub>3</sub>	20.0	13	>1,200	<sup>13</sup> 7.5	>1,200	<sup>4</sup> 4	>1,200	<sup>4</sup> 2.5	>1,200	<sup>2</sup> 2	-	-	-	-
CuCl	25.0	12	425	<sup>4</sup> 2.5	400	<sup>4</sup> 1	400	<sup>4</sup> 0.75	462	<sup>4</sup> 0.5	-	-	-	-
CuFeS <sub>2</sub>	25.0	13	910	<sup>5</sup> 0.75	930	<sup>6</sup> 0.33	>1,150	<sup>2</sup> 0.25	>1,200	<sup>2</sup> 0.13	-	-	-	-
CuO	25.0	8	1,083	<sup>2</sup> 1.5	1,088	<sup>2</sup> 0.25	1,088	<sup>2</sup> 0.5	>1,200	<sup>2</sup> 0.25	-	-	-	-
CuS	26.1	14	443	<sup>5</sup> 6	573	<sup>5</sup> 1	-	-	-	-	-	-	-	-
FeCl <sub>3</sub>	15.0	13	305	<sup>7</sup> 5.75	300	<sup>7</sup> 4.5	-	-	-	-	-	-	-	-
FeCr <sub>2</sub> O <sub>4</sub>	30.0	12	110	7	1,200	<sup>2</sup> 6.25	>1,200	<sup>2</sup> 3.25	>1,200	<sup>2</sup> 2	-	-	-	-
Fe <sub>2</sub> O <sub>3</sub>	12.0	14	60	7	87	7	101	7	130	7	276	7	1,097	<sup>6</sup> 0.83
Fe <sub>3</sub> O <sub>4</sub>	25.0	9	1,118	7	1,144	<sup>2</sup> 3.5	1,123	<sup>3</sup> 3	905	<sup>6</sup> 1	360	<sup>6</sup> 0.5	446	<sup>6</sup> 0.25
FeS <sub>2</sub>	20.0	12	698	5.5	808	7	700	<sup>6</sup> 0.75	-	-	-	-	-	-
FeSO <sub>4</sub> •7H <sub>2</sub> O	12.0	13	113	<sup>5</sup> 6.25	118	<sup>5</sup> 5.5	118	<sup>5</sup> 3	123	<sup>5</sup> 1.75	292	7	>1,200	<sup>2</sup> 0.67
H <sub>2</sub> SO <sub>4</sub>	26.3	14	317	<sup>7</sup> 5.5	321	<sup>7</sup> 3	-	-	-	-	-	-	-	-
KCl	17.4	14	42	7	59	7	86	7	112	<sup>6</sup> 6.5	90	<sup>6</sup> 2.5	93	<sup>6</sup> 2.25
NaCl	20.0	12	39	7	68	7	70	7	60	<sup>3</sup> 0.5	-	-	-	-
NiS	34.2	14	268	7	373	7	322	7	330	7	-	-	-	-
PbS	50.0	11	935	<sup>6</sup> 0.4	984	<sup>6</sup> 0.25	-	-	-	-	-	-	-	-
SiC	20.0	12	256	7	398	7	>1,200	<sup>2</sup> 4.75	>1,200	<sup>3</sup> 3	-	-	-	-
SiO <sub>2</sub>	25.0	14	33	7	44	7	55	7	73	7	141	7	142	7
TiO <sub>2</sub>	10.0	13	67	7	109	7	150	7	228	7	>1,200	<sup>2</sup> 2.67	>1,200	<sup>2</sup> 1.25
ZnCl <sub>2</sub>	14.0	<sup>8</sup> 9	728	<sup>7</sup> 4.25	723	<sup>7</sup> 1.25	730	<sup>7</sup> 0.75	593	<sup>6</sup> 0.5	-	-	-	-
ZnO	9.5	14	87	7	128	7	184	7	301	<sup>5</sup> 5.75	>1,200	<sup>2</sup> 1.75	>1,200	<sup>2</sup> 1.25
ZnS	30.0	12	139	7	236	7	>1,200	<sup>6</sup> 6	>1,200	<sup>3</sup> 3.75	>1,200	<sup>2</sup> 2.25	>1,200	<sup>2</sup> 2

<sup>1</sup>Maximum temperature obtained in the indicated time interval. A maximum time interval of 7 min was arbitrarily chosen.<sup>2</sup>Test terminated: temperature approached or exceeded the thermocouple limitation. Sheath material melts at approximately 1,200 °C.<sup>3</sup>Test terminated: the quartz, Teflon, or firebrick components of the test cell exhibited incipient absorption of microwaves at higher power.<sup>4</sup>Test terminated: sample melted.<sup>5</sup>Test terminated: temperature began decreasing at the indicated time.<sup>6</sup>Test terminated: sample arced.<sup>7</sup>Test terminated at sample boiling point.<sup>8</sup>Smaller volume used because sample violently boiled.

NOTE.—Dashes indicate no test. Sample test cell not compatible with this sample at high power because incipient heating was previously observed at lower power.

## VARIABLE POWER STUDY

The data in table 3 show that incident microwave power level does have an effect on the heating rates of many chemicals and minerals. For the most part, the heating rates increased as the power was increased except in cases of highly absorbing materials or microwave-transparent materials, which showed very little change with power. Some compounds that did not heat at low power showed drastic changes with an increase of microwave power. The heating-rate profiles for each of the chemicals and minerals tested at varied power levels are included in appendix C. The curves are scaled duplications of the analog strip-chart recordings, and depict a variety of heating-rate responses that are characteristic of the materials.

Some of the oxides tested,  $\text{Al}_2\text{O}_3$ ,  $\text{Fe}_2\text{O}_3$ ,  $\text{TiO}_2$ , and  $\text{ZnO}$ , showed steady increases in heating rates with increasing power levels. Each of these samples attained a different maximum temperature dependent on the input power. Both  $\text{CuO}$  and  $\text{Fe}_3\text{O}_4$  (magnetite) are extremely good microwave absorbers, reaching temperatures in excess of  $1,000^\circ\text{C}$  in less than 1 min. The  $\text{CuO}$  (appendix C) reached a maximum temperature six times faster when irradiated at 1,000 W in comparison with an incident power of 500 W.

The  $\text{Fe}_3\text{O}_4$  (appendix C) sample heated to a maximum temperature with a negligible rate change regardless of input power, heating to about  $900^\circ\text{C}$  at 1 min, and slowly increasing for the duration of the test. According to the heating-rate curves for  $\text{Fe}_3\text{O}_4$ , a power level of 500 W should be as effective as 1,000 or 2,000 W to obtain the maximum heating rate when 25 g of  $\text{Fe}_3\text{O}_4$  is microwaved. Magnetite is a very good absorber that can promote heating when added to materials that are not microwave absorbers. This use of magnetite can significantly broaden the range of potential applications for heating of ores and concentrates. Microwave heating of a cinnabar concentrate was feasible for extracting the mercury when magnetite was added to promote heating. Although little or no microwave heating was obtained with  $\text{Hg}$ ,  $\text{HgS}$ , or the concentrate, adding 20 pct magnetite to the mercury concentrate resulted in a sample temperature above  $600^\circ\text{C}$ . This was hot enough to vaporize mercury from the sample. In an oxygen atmosphere, a maximum temperature of  $688^\circ\text{C}$  was obtained, and mercury droplets condensed within the test cell. The amount of liberated mercury was considerably less in a helium atmosphere because the sublimation temperature of  $\text{HgS}$  ( $583^\circ\text{C}$ ) was not obtained. Higher temperature and better mercury recovery with the oxygen atmosphere were attributed to oxidation of free sulfur, which was produced when the  $\text{HgS}$  decomposed. By increasing either the amount of magnetite or the incident power level, the oxygen can be eliminated, thereby improving mercury recovery without producing  $\text{SO}_2$ .

At 500-W input power,  $\text{Cr}_2\text{O}_3$  (appendix C) and  $\text{FeCr}_2\text{O}_4$  (appendix C) heated to no more than  $110^\circ\text{C}$  in 7 min. With an increase in power to 1,000 W both samples slowly heated to a temperature of about  $100^\circ\text{C}$  followed by rapid heating to greater than  $1,200^\circ\text{C}$  at 4 and 6.25 min, respectively. When the 500-W test for  $\text{Cr}_2\text{O}_3$  was arbitrarily extended, the sample reached a critical temperature of  $100^\circ\text{C}$  at about 12 min and was followed by the same rapid heating (runaway effect) observed in the higher power tests. At the critical temperature, it appears that a change in the dielectric properties enabled the sample to readily absorb the available power, resulting in a sudden and dramatic rise in temperature.

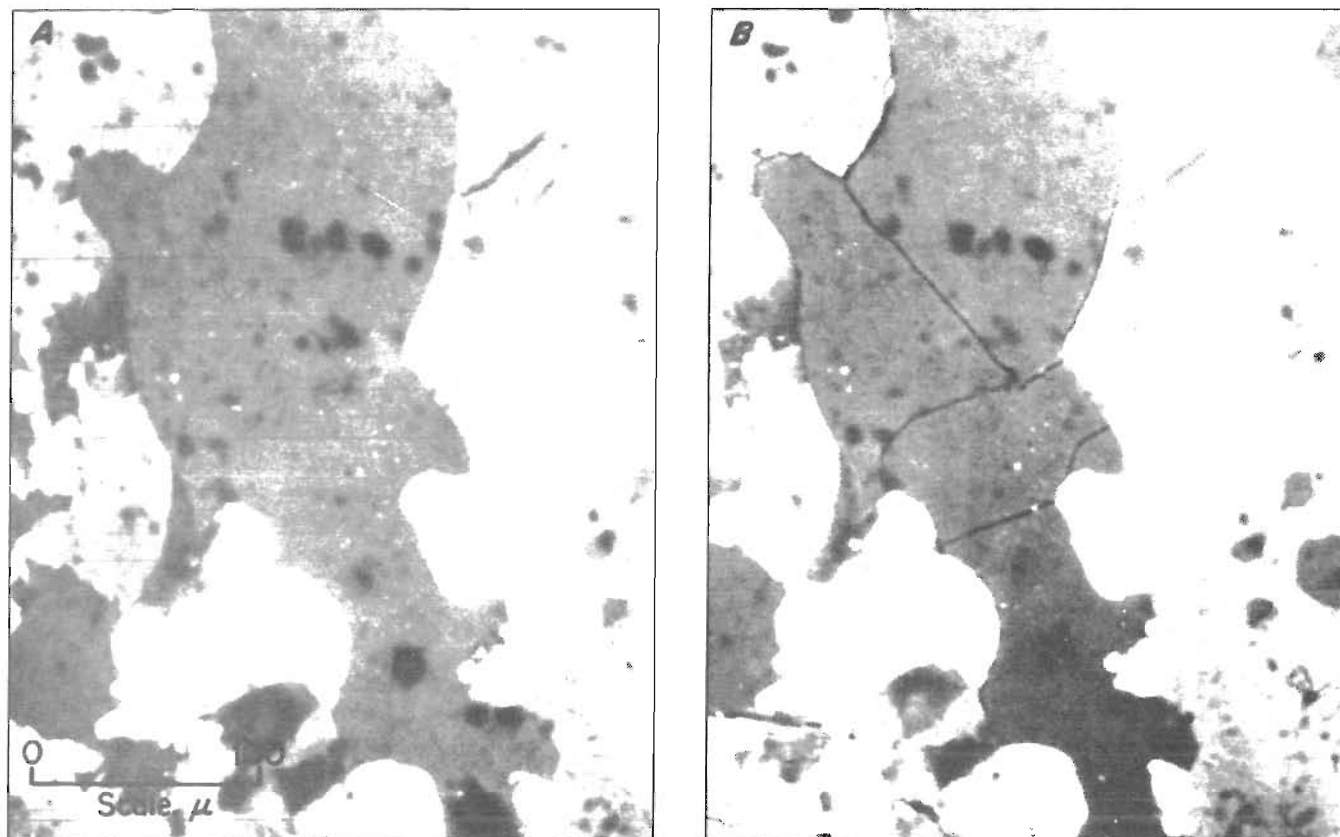
The  $\text{SiO}_2$  (appendix C) and  $\text{CaCO}_3$  (appendix C), which are common gangue constituents, were poor absorbers. The slight increase in the observed temperature as power was increased was due to oven heating effects from a lack of an absorbing load. In these situations the percentage for error in the sample temperature measurements increases and it is believed that there is no significant increase in heating rates with increasing input power levels for these low-absorbing samples.

When a highly absorbing mineral is contained in a microwave-transparent matrix such as  $\text{SiO}_2$  or  $\text{CaCO}_3$ , stress fracturing along and through the grain boundaries will occur when the material is microwaved. This in turn will lead to a reduction in energy consumption and increased mineral liberation during comminution. Stress cracking is readily observable in figure 3. The sample, before and after microwaving, is a taconite ore. The lighter phases are iron-bearing minerals and the darkest phase is a siliceous gangue. Reductions of grinding energy of up to 24 pct were obtained with taconite ores containing highly receptive  $\text{Fe}_3\text{O}_4$  in an  $\text{SiO}_2$  matrix.

The  $\text{SiC}$  material (appendix C), is an example of a good absorber at high powers that showed a dramatic effect of power on its heating rate. After 7 min at 500 and 1,000 W,  $\text{SiC}$  heated to  $256^\circ$  and  $398^\circ\text{C}$ , respectively. When the power was increased to 1,500 W, the material heated more quickly to a critical temperature of  $600^\circ\text{C}$  where a runaway effect was observed. Because of its heating properties,  $\text{SiC}$  has been used commercially to line microwave-transparent chambers that can be placed inside a microwave oven. The  $\text{SiC}$  absorbs the microwave energy and conducts heat to the sample within the chamber.

Three of the chlorides tested,  $\text{CuCl}$ ,  $\text{ZnCl}_2$ , and  $\text{FeCl}_3$ , heated faster to their respective maximum temperatures as the power was increased. At an input power of 500 W,  $\text{CuCl}$  (appendix C) reached  $425^\circ\text{C}$  in 3 min and showed signs of melting. The melting point of  $\text{CuCl}$ ,  $430^\circ\text{C}$ , was attained in successively less time as power was increased, finally showing incipient melting after 30 s at 2,000 W. For  $\text{ZnCl}_2$  (appendix C), an increase in power reduced the time needed to reach its boiling point ( $732^\circ\text{C}$ ) from

Figure 3



*Microwave-induced stress cracking in a taconite (iron ore) sample. A, Before; B, after.*

4.25 min at 500 W to 45 s at 1,500 W. At 500 and 1,000 W,  $\text{FeCl}_3$  (appendix C) heated slowly to about 60 °C and exhibited mild runaway heating to its melting point of 300 °C, at which point the test was terminated.

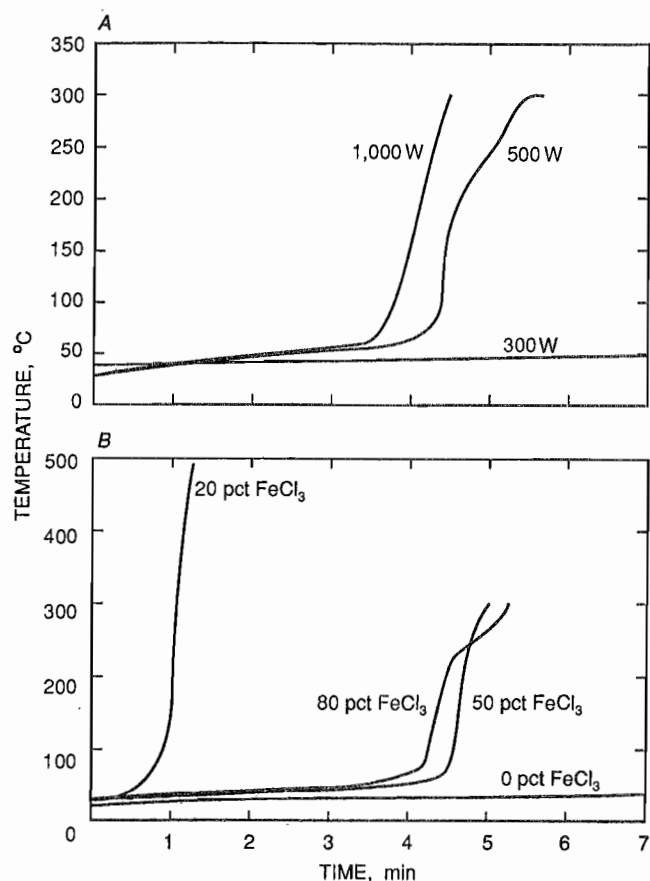
Additional tests were conducted with  $\text{FeCl}_3$  at 200 and 300 W in response to a leaching application that resulted in an unexpectedly high sample temperature of nearly 1,200 °C. Two chlorides,  $\text{FeCl}_3$  and  $\text{NaCl}$  (appendix C), were mixed with a chalcopyrite ( $\text{CuFeS}_2$ ) ore and microwaved, resulting in copper extraction greater than 99 pct after water leaching the mixture. Less than 10-pct copper recovery was obtained with the same mixture that was not microwaved. Although pure chalcopyrite (appendix C) heats well, the ore containing a small percentage of chalcopyrite heated to less than 100 °C. The same ore with additions of either  $\text{FeCl}_3$  or  $\text{NaCl}$  did not exceed 100 °C when microwaved. However, with additions of both  $\text{FeCl}_3$  and  $\text{NaCl}$ , the ore heated rapidly to 1,190 °C. Further tests were conducted to try to explain this phenomenon. Dry mixtures of  $\text{FeCl}_3$  and  $\text{NaCl}$  were microwaved at 300 W, which was approximately the power level at which the ore-salt mixture heated to 1,190 °C. Baseline heating rates for each of the salts (figure 4) showed that neither

salt heated appreciably at 300 W. However, with mixtures of 50 and 80 pct  $\text{FeCl}_3$  and the remainder  $\text{NaCl}$ , mild runaway heating was observed, and with 20 pct  $\text{FeCl}_3$  the mixture quickly heated to almost 500 °C in just over 1 min. Similar results were obtained when  $\text{CaCl}_2$  and  $\text{KCl}$  were mixed with  $\text{FeCl}_3$ . The mechanism by which these mixed salts promote microwave heating is not fully understood.

The only liquid tested at varied power levels was concentrated  $\text{H}_2\text{SO}_4$  (appendix C), containing 2 to 5 pct water. The  $\text{H}_2\text{SO}_4$  reached its boiling point of 338 °C within 5.5 min at 500 W, and 3 min at 1,000 W. The use of  $\text{H}_2\text{SO}_4$  as an additive to improve metal extraction from various gold and silver ores prior to roasting was demonstrated. When sulfide ores were mixed with  $\text{H}_2\text{SO}_4$  and microwave roasted followed by conventional cyanidation, 70- to 80-pct metal extraction was obtained. With no  $\text{H}_2\text{SO}_4$ /microwave pretreatment, 50-pct metal extraction was obtained. The increased extraction was comparable with those observed with conventional heating.

All of the sulfides tested showed an increase in heating rates as power was increased. The  $\text{FeS}_2$  (appendix C) quickly heated to over 600 °C in less than 2 min at power levels of 500, 1,000, and 1,500 W. With an input power of

Figure 4



Microwave heating-rate curves. A,  $\text{FeCl}_3$ ; B, mixtures of  $\text{FeCl}_3$  and  $\text{NaCl}$  irradiated at 300 W.

500 or 1,000 W the samples remained at a fairly constant temperature between 2 and 7 min during which time sulfur was observed escaping from the samples. At 1,500 W the sample quickly reached 700 °C in 45 s and arced violently.

At 2,000 W the sample arced within a few seconds causing termination of the test. When pyrite is heated to between 600° and 800 °C, a chemical change occurs resulting in the formation of the magnetic mineral pyrrhotite, as reported by Hall and Finch (10) and Jacobs and others (11). With microwave treatment, pyrite that contained less than a 1-pct magnetic fraction was converted to pyrrhotite having a 98-pct magnetic fraction. Rapid and selective heating of an ore containing pyrite may lead to a process for the removal of iron and sulfur impurities via magnetic separation. Microwave conversion of pyrite to magnetic pyrrhotite may also be used in removing sulfur from pyritic coal.

Microwave-induced carbothermic reductions of several oxides produced the metals iron, lead, and bismuth. Because powdered carbon (amorphous and graphite) readily heats (table 1), it served a dual purpose as a microwave absorber to promote heating and as a reductant to produce metal. A taconite ore containing 39 pct iron as magnetite was mixed with powdered carbon and heated with microwaves under an argon atmosphere. Temperatures greater than 1,200 °C were obtained and a sponge iron containing 85 pct iron was produced. Another taconite and carbon mixture was covered with a slag forming layer of lime and silica. After holding the mixture at approximately 950 °C for 40 min, the microwave power was raised to 3,000 W for 5 min to fuse the sample. A nonmagnetic vitreous slag was formed and an iron bead containing more than 99 pct iron was recovered. During these preliminary tests, about 42 pct of the iron in the original taconite ore was recovered in the iron bead. Lead and bismuth oxides were similarly reduced to metal with carbon, but without the addition of lime and silica. For metals with very high melting points such as titanium and zirconium, an additional source of heat may be required to keep the metal product from freezing since the newly formed metal is not heated by microwaves.

## SUMMARY

Temperatures ( $\pm 2$  pct) and rates of heating were determined for selected minerals and chemicals during microwave irradiation at 2,450 MHz for incident power levels of less than 500 W and up to 3,000 W. Many minerals of value heated, whereas most of the gangue minerals did not heat. In general, an increase in power increased the heating rates except for very poor absorbing materials such as  $\text{CaCO}_3$  and  $\text{SiO}_2$ , which did not heat well at any of the power levels tested, or for very good absorbing materials such as  $\text{CuO}$ ,  $\text{Fe}_3\text{O}_4$ , and  $\text{PbS}$ , which heated rapidly at all power levels tested.

Using the heating data, several applications of microwave energy in mineral processing were demonstrated in the laboratory. Microwave-induced, high-temperature carbothermic reductions of metal oxide compounds produced metal beads. Gold, silver, and copper ores were microwave roasted resulting in improved metal extraction comparable with those observed with conventional heating. Pyrite was converted to magnetic pyrrhotite to demonstrate that pyrite as an impurity could be removed from an ore or from high-sulfur coal.



The identification of magnetite and powdered carbon as excellent absorbers of microwaves resulted in novel applications of extractive metallurgy. Doping a poor microwave absorber, cinnabar, with magnetite resulted in a

temperature sufficient to volatilize mercury. The efficiency of grinding for several iron ores containing magnetite was increased due to rapid microwave heating that induced stress cracking.

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## APPENDIX A.—DESCRIPTIVE LIST OF THE MICROWAVED SAMPLES

Chemical	Source	Grade	Purity, pct
AgBr	Fisher Scientific Co.	Purified	-
AgCl	Mallinrodt	Analytical reagent	99
AgI	Fisher Scientific Co.	Purified	-
Ag <sub>2</sub> S	Cooper Chemical Co.	.. do.	-
Al	Alfa Products	-	99.9
AlCl <sub>3</sub>	B&A Chemicals	Reagent	99
AlCl <sub>3</sub> •6H <sub>2</sub> O	Mallinrodt Chemical Works	Analytical reagent	99
Al(ClO <sub>3</sub> ) <sub>3</sub> •6H <sub>2</sub> O	.. do.	.. do.	99
Al <sub>2</sub> O <sub>3</sub>	Alfa Products	-	96
Al <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> •18H <sub>2</sub> O	J.T. Baker Chemical Co.	Analytical reagent	99.6
Al <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> •K <sub>2</sub> SO <sub>4</sub> •24H <sub>2</sub> O	B&A Chemicals	Reagent ACS	99.5
BaCl <sub>2</sub>	Fisher Scientific Co.	Certified	99
BaCl <sub>2</sub> •2H <sub>2</sub> O	J.T. Baker Chemical Co.	Analytical reagent	99
BaS	Matheson, Coleman, and Bell (MC/B)	Purified	-
C (amorphous, <1 μm)	Leco Corp.	-	99
C (graphite, <1 μm)	Carbone Corp.	Ultra-F	99.9
C (graphite, -200 mesh)	.. do.	.. do.	99.9
CaBr <sub>2</sub> •2H <sub>2</sub> O	J.T. Baker Chemical Co.	Purified	98
CaCl <sub>2</sub>	Mallinrodt Chemical Works	Analytical reagent	96
CaCl <sub>2</sub> •2H <sub>2</sub> O	J.T. Baker Chemical Co.	.. do.	99
CaCO <sub>3</sub>	B&A Chemicals	Reagent ACS	99
CaF <sub>2</sub>	J.T. Baker Chemical Co.	Analytical reagent	98.6
CaO	.. do.	.. do.	98
CaS	Fisher Scientific Co.	Purified	-
CCl <sub>4</sub>	J.T. Baker Chemical Co.	Analytical reagent	99
CdCl <sub>2</sub>	.. do.	.. do.	99.4
CdCl <sub>2</sub> •2.5H <sub>2</sub> O	Mallinrodt Chemical Works	.. do.	99
CdS	Fisher Scientific Co.	Certified	99
CeCl <sub>3</sub>	Alfa Products	-	99.9
CeCl <sub>3</sub> •7H <sub>2</sub> O	.. do.	-	99.9
CeO <sub>2</sub>	Lindsay Chemical Co.	-	99.9
Co	Belmont Chemical Inc.	-	99.9
CoCl <sub>2</sub>	B&A Chemicals	Reagent	99.0
CoCl <sub>2</sub> •6H <sub>2</sub> O	J.T. Baker Chemical Co.	Purified	-
Co <sub>2</sub> O <sub>3</sub>	BKH Co.	USP	99
CoS	Hudson Laboratories	-	99.9
CrCl <sub>3</sub>	Fisher Scientific Co.	Purified	-
CrCl <sub>3</sub> •6H <sub>2</sub> O	J.T. Baker Chemical Co.	Analytical reagent	99
CrO <sub>3</sub>	B&A Chemicals	Reagent ACS	99
Cr <sub>2</sub> O <sub>3</sub>	J.T. Baker Chemical Co.	Chemically pure	-
Cu	B&A Chemicals	Purified	99
CuBr	Fisher Scientific Co.	Certified	99
CuBr <sub>2</sub>	J.T. Baker Chemical Co.	Analytical reagent	98
CuCl	Alfa Products	-	99
CuCl <sub>2</sub> •2H <sub>2</sub> O	Mallinrodt Chemical Works	Analytical reagent	99
CuF <sub>2</sub> •2H <sub>2</sub> O	B&A Chemicals	Technical	-
CuO	.. do.	Reagent	99
Cu <sub>2</sub> O	J.T. Baker Chemical Co.	Analytical reagent	99.5
CuS	Fisher Scientific Co.	Certified	95.6
Fe	J.T. Baker Chemical Co.	Purified	-
FeBr <sub>2</sub> •6H <sub>2</sub> O	Alfa Products	-	99
FeCl <sub>2</sub>	.. do.	-	99
FeCl <sub>2</sub> •2H <sub>2</sub> O	Fisher Scientific Co.	Certified	99
FeCl <sub>2</sub> •4H <sub>2</sub> O	J.T. Baker Chemical Co.	Analytical reagent	99
FeCl <sub>3</sub>	Fisher Scientific Co.	Purified	-
FeCl <sub>3</sub> •6H <sub>2</sub> O	B&A Chemicals	Reagent ACS	99.5
Fe <sub>2</sub> O <sub>3</sub>	J.T. Baker Chemical Co.	Analytical reagent	98
FeSO <sub>4</sub> •7H <sub>2</sub> O	Mallinrodt Chemical Works	.. do.	99

See notes at end of table.

<u>Chemical</u>	<u>Source</u>	<u>Grade</u>	<u>Purity, pct</u>
Fe <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> •9H <sub>2</sub> O	Eastman Kodak Co.	Reagent	-
Hg	J.T. Baker Chemical Co.	Analytical reagent	99.99
HgCl <sub>2</sub>	.. do.	.. do.	99.5
HgS	Fisher Scientific Co.	Purified	-
H <sub>2</sub> SO <sub>4</sub>	Mallincrodt Chemical Works	Analytical reagent	95.8
KBr	B&A Chemicals	Reagent ACS	-
KCl	Fisher Scientific Co.	Purified	-
LaCl <sub>3</sub>	Alfa Products	-	99
LaCl <sub>3</sub> •7H <sub>2</sub> O	American Potash and Chemicals	-	99.99
La <sub>2</sub> O <sub>3</sub>	Source unknown/in-house analysis	-	99
LiBr•H <sub>2</sub> O	Aldrich Chemical Co.	-	99
LiCl	J.T. Baker Chemical Co.	Analytical reagent	99
Mg	B&A Chemicals	Reagent	-
MgBr <sub>2</sub> •6H <sub>2</sub> O	EM Industries	Guaranteed reagent	-
MgCl <sub>2</sub> •6H <sub>2</sub> O	Spectrum Chemicals	Reagent ACS	99
MgO	BKH Co.	USP	99
MgSO <sub>4</sub> •7H <sub>2</sub> O	Mallincrodt Chemical Works	Analytical reagent	99
MnBr <sub>2</sub> •4H <sub>2</sub> O	Alfa Products	-	99
MnCl <sub>2</sub>	.. do.	-	99
MnCl <sub>2</sub> •4H <sub>2</sub> O	J.T. Baker Chemical Co.	Analytical reagent	99.0
MnO <sub>2</sub>	B&A Chemicals	.. do.	99.5
MnSO <sub>4</sub> •H <sub>2</sub> O	J.T. Baker Chemical Co.	.. do.	99
Mo	Fansteel Metallurgical Corp.	-	99
MoO <sub>3</sub>	B&A Chemicals	Analytical reagent	99.5
NaBr	Mallincrodt Chemical Works	.. do.	99
NaCl	VWR Scientific	Reagent ACS	-
Na <sub>2</sub> MoO <sub>4</sub>	Fisher Scientific Co.	Purified	-
Na <sub>2</sub> SO <sub>4</sub>	J.T. Baker Chemical Co.	Analytical reagent	99
Nb	Kawecki Chemical Co.	Purified	99
Nb <sub>2</sub> O <sub>5</sub>	.. do.	.. do.	99
NH <sub>4</sub> Cl	J.T. Baker Chemical Co.	Analytical reagent	99.6
5(NH <sub>4</sub> ) <sub>2</sub> •12WO <sub>3</sub>	Source unknown/in-house analysis	-	95
Ni	Alfa Products	-	99
NiBr <sub>2</sub> •2H <sub>2</sub> O	.. do.	-	99
NiCl <sub>2</sub>	B&A Chemicals	Reagent	99
NiCl <sub>2</sub> •6H <sub>2</sub> O	Matheson, Coleman, and Bell (MC/B)	.. do.	99
NiF <sub>2</sub> •4H <sub>2</sub> O	B&A Chemicals	Technical	-
NiO	J.T. Baker Chemical Co.	Analytical reagent	99
NiS	Hudson Laboratories	-	99.9
Pb	J.T. Baker Chemical Co.	Analytical reagent	99.9
PbBr <sub>2</sub>	Alfa Products	-	95
PbCl <sub>2</sub>	J.T. Baker Chemical Co.	Analytical reagent	99
PbO	Mallincrodt Chemical Works	.. do.	99
PbS	Matheson, Coleman, and Bell (MC/B)	Technical	-
PrCl <sub>3</sub>	Alfa Products	-	99.9
S	Matheson, Coleman, and Bell (MC/B)	Reagent ACS	99
Sb	.. do.	-	99.5
SbCl <sub>3</sub>	J.T. Baker Chemical Co.	Analytical reagent	99
SbCl <sub>5</sub>	.. do.	.. do.	99
SiC	Buehler Ltd.	-	-
Sn	J.T. Baker Chemical Co.	Analytical reagent	99.9
SnBr <sub>2</sub>	Alfa Products	-	99
SnCl <sub>2</sub>	B&A Chemicals	Reagent ACS	95
SnCl <sub>2</sub> •2H <sub>2</sub> O	Mallincrodt Chemical Works	Analytical reagent	99
SnCl <sub>4</sub>	J.T. Baker Chemical Co.	.. do.	99.4
SnCl <sub>4</sub> •5H <sub>2</sub> O	.. do.	.. do.	99
SrCl <sub>2</sub> •6H <sub>2</sub> O	Matheson, Coleman, and Bell (MC/B)	Reagent ACS	99
Ta	Kawecki Chemical Co.	Purified	99
TiB <sub>2</sub>	Matheson, Coleman, and Bell (MC/B)	Reagent	99
TiCl <sub>4</sub>	J.T. Baker Chemical Co.	Purified	-
TiO <sub>2</sub>	.. do.	Analytical reagent	99
V	Vanadium Corporation of America	-	99.5

See notes at end of table.

<u>Chemical</u>	<u>Source</u>	<u>Grade</u>	<u>Purity, pct</u>
V <sub>2</sub> O <sub>5</sub> .....	Matheson, Coleman, and Bell (MC/B) ..	Technical .....	-
YCl <sub>3</sub> .....	Electronic Space Products Ind. ....	-	99.9
Y <sub>2</sub> O <sub>3</sub> .....	Source unknown/in-house analysis ....	-	99.9
W .....	Sylvania Electric Products .....	-	99
WCl <sub>6</sub> .....	Alfa Products .....	-	99
WO <sub>3</sub> .....	Johnson Matthey/Aesar Group .....	Analytical reagent .....	99.8
Zn .....	J.T. Baker Chemical Co. ....	.. do. ....	99
ZnBr <sub>2</sub> .....	Fisher Scientific Co. ....	Certified .....	99
ZnCl <sub>2</sub> .....	J.T. Baker Chemical Co. ....	Analytical reagent .....	99.2
ZnO .....	Mallinckrodt Chemical Works .....	.. do. ....	99
ZnS .....	Fisher Scientific Co. ....	Purified .....	-
Zr .....	Wah Chang, Albany .....	.. do. ....	-
ZrO <sub>2</sub> .....	Fisher Scientific Co. ....	.. do. ....	-

ACS America Chemical Society specifications.

USP United States Pharmacopeia specifications.

NOTE.—Dashes (-) indicate that information was not reported on label by manufacturer.

**APPENDIX B.—TYLER SIEVE SCREEN ANALYSES OF CHEMICALS AND MINERALS  
MICROWAVED IN THE VARIED POWER LEVEL STUDY, PERCENT**

Material <sup>1</sup>	Size fractions, mesh				
	+ 65	-65 + 100	-100 + 200	-200 + 325	-325
Al <sub>2</sub> O <sub>3</sub> .....	0.5	0.8	15.7	30.8	52.2
CaCl <sub>2</sub> .....	37.3	11.3	20.8	14.6	16.0
CaCO <sub>3</sub> .....	0.0	0.0	0.0	7.3	92.7
Cr <sub>2</sub> O <sub>3</sub> .....	2.2	16.9	73.5	5.5	1.9
CuCl .....	15.9	2.7	17.6	19.8	44.0
CuFeS <sub>2</sub> .....	6.6	4.2	13.0	11.7	64.5
CuO .....	0.3	0.7	8.6	17.7	72.7
CuS .....	0.0	0.0	1.9	5.6	92.5
FeCl <sub>3</sub> .....	1.9	2.0	13.5	32.6	50.0
FeCr <sub>2</sub> O <sub>4</sub> .....	0.0	0.0	35.2	26.1	38.7
Fe <sub>2</sub> O <sub>3</sub> <sup>2</sup> .....	0.0	4.0	2.3	1.9	91.8
Fe <sub>3</sub> O <sub>4</sub> .....	19.6	42.2	36.9	1.2	0.1
FeS <sub>2</sub> .....	30.4	5.3	6.3	3.5	54.5
FeSO <sub>4</sub> ·7H <sub>2</sub> O .....	97.2	1.4	0.9	0.3	0.2
KCl .....	40.2	38.7	19.7	1.3	0.1
NaCl .....	89.0	9.5	1.5	0.0	0.0
NiS .....	98.8	0.3	0.3	0.3	0.3
PbS .....	4.7	7.3	15.3	12.0	60.7
SiC .....	0.0	0.0	0.0	1.4	98.6
SiO <sub>2</sub> .....	58.6	30.3	11.0	0.1	0.0
TiO <sub>2</sub> <sup>2</sup> .....	0.0	0.0	4.0	2.0	94.0
ZnCl <sub>2</sub> .....	95.8	4.2	0.0	0.0	0.0
ZnO .....	2.5	9.2	29.2	48.7	10.4
ZnS .....	4.0	7.2	32.2	19.2	37.3

<sup>1</sup>Dry screened unless otherwise noted.

<sup>2</sup>Wet screened.

APPENDIX C.—HEATING-RATE CURVES OF SAMPLES MICROWAVED  
IN THE VARIED POWER LEVEL STUDY

